Anomalies in Polymer-Based Helium Leak Standards

Leak detection is required to maintain the quality of many industrial products including containers for nuclear waste, food packaging, hermetically sealed medical devices (such as pace makers), and electronics for space applications. Users of these products require accurate measurements of gas leak rates both into and out of sealed containers. Helium "leak" artifacts provide a steady flow of helium that is used to calibrate helium leak detectors, which, in turn, are used to measure the amount of gas that leaks in or out of a sealed package. Most helium leak artifacts exploit the permeability of fragile glasses to helium. NIST calibrates glass helium leak artifacts (i.e., measures their gas flow) as a function of temperature between 0 °C and 50 °C and provides an expanded uncertainty (k=2) of 1 % to 3 %.

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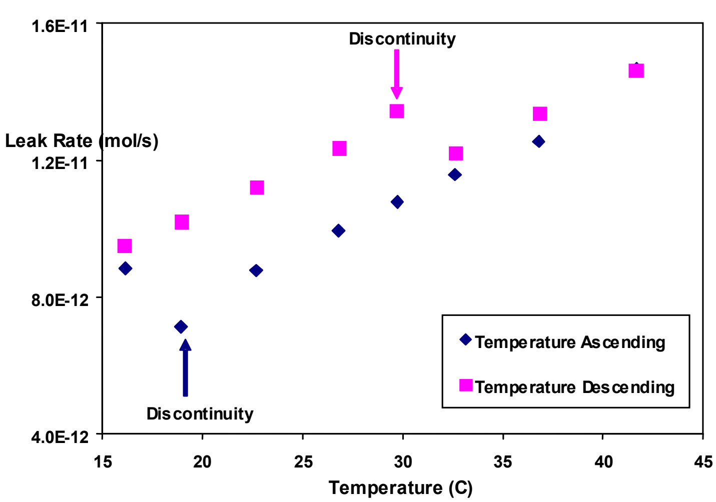
A different type of helium leak artifact that uses break-resistant Teflon as the permeation element was sent to NIST for calibration. Our calibration of this artifact showed severe hysteresis and other anomalies in the measured leak rate that exceeded NIST's measurement uncertainties. We followed up this observation by obtaining Teflon-based leak artifacts from several different manufacturers and measuring the stability of their helium leak rates to identify possible anomalous behaviors that might jeopardize the accuracy of customer calibrations. Anomalies of up to 30 % in the measured helium leak rate at a give temperature were observed with one artifact. The graph below shows a calibration of a Teflon-based permeation leak artifact. The calibration started at 16 °C and ascended in temperature to 42 °C (blue symbols); the data were repeated as the temperature descended (pink symbols). Note the hysteresis between 19 °C and 33 °C. The arrows indicate the temperatures where known phase transitions occur in this type of Teflon.



These anomalies probably result from phase transitions in Teflon's crystal-structure near 19 °C and 30 °C. Interestingly, an exceptional NIST scientist, George Furikawa, was a pioneer in measuring these phase changes in Teflon (PTFE) over 50 years ago.

Typically, manufacturers and secondary calibration laboratories calibrate a helium leak artifact at one temperature, or a narrow range of temperatures (*e.g.*, 23 °C ± 5 °C). They may give the customer a "temperature coefficient" to correct for the difference between the calibration temperature and the temperature at which the artifact is used. With such a protocol, the anomalous behavior of Teflon-based leaks might not be noticed. NIST routinely calibrates leak artifacts as a function of temperature between 0 °C and 50 °C. This more-demanding procedure did detect the anomalies. These results were presented at the American Vacuum Society's 52nd International Symposium & Exhibition,

NIST calibrations and research will enable secondary calibration laboratories and customers to make an informed choice between stable, but fragile, glass-based leak standards and more rugged but less stable Teflon-based standards.

Future Plans: NIST is testing other polymer leak elements of different compositions to see if these are suitable to replace Teflon, thereby enabling a more rugged (i.e., unbreakable) leak artifact without the anomalous behavior. NIST can use the same measurement techniques to measure the permeation properties of many industrially relevant materials, and is considering further testing in order to make this a commercially offered service.

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